

NONMETALLIC INCLUSIONS IN STEEL

Part I. Products of Deoxidation

By Stephen F. Urban and John Chipman



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Abstract

Part I contains a description of the nonmetallic inclusions in steels prepared by the addition of deoxidizing elements to liquid iron saturated with oxygen. The effects of heat treatment and of forging upon the structure of the inclusions is described.

The ore microscope has been used to study inclusions in a polished specimen by the aid of polarized light. A new technique has been developed by means of which individual small inclusions are removed from the steel and their refractive indices measured. Both of these methods have proved useful in the identification of certain types of inclusions.

Subsequent parts will deal with sulphide, oxide-sulphide, and nitride inclusions and with etching methods for their identification.

IN tracing the origin of nonmetallic inclusions in steel and in studying methods for their control in steel making practice, it is often a matter of importance to be able to determine the nature of the individual inclusions. This task is rendered difficult by the small size of the majority of these bodies and the consequent inapplicability of the customary quantitative methods. An approach toward a solution of this problem may be obtained through the microscopic study of inclusions in steels prepared under such carefully controlled conditions that each heat contains nonmetallic matter of a single and definitely known type. On the basis of such studies the microscopist is able to identify many of the common inclusion types encountered in commercial steels. The application of petrographic and qualitative micro-chemical methods improves the reliability of the identification and increases the variety of types which may be identified.

This paper is taken from a dissertation submitted by S. F. Urban in partial fulfilment of the requirements for the degree of Doctor of Science at the University of Michigan. Dr. Urban is now associated with the metallurgical department of the National Tube Company at Ellwood City, Pa. Dr. Chipman is research engineer, department of engineering research, University of Michigan, Ann Arbor, Mich. Manuscript received July 2, 1934.

PREPARATION OF KNOWN OXIDE INCLUSIONS

Matveieff (1)¹ prepared synthetic inclusions by forging an iron pipe containing known nonmetallic materials. Very pure inclusions were prepared by Wohrman (2). He packed an electrolytic iron crucible with known oxides, placed it in an alundum crucible and melted in an Arsem furnace. This arrangement eliminated most of the objectionable fluxing of the crucible by the inclusions. Herty and his co-workers (3, 4) added deoxidizers to successive ladles of metal from an oxidized bath in an electric furnace. Benedicks and Löfquist (5) have depended upon commercial steels, slags, and minerals. Important microscopic studies of inclusions in commercial steels have recently been reported by Grant (6), Sims and Lilliequist (7), Zieler (8) and Löfquist (9). For the present study the melts were prepared in a small magnesia-lined induction furnace which was capable of rapidly melting a charge of three or four kilograms. The charge consisted of electrolytic or ingot iron with sufficient iron oxide to saturate the liquid metal. When the melt was thoroughly liquid it was held at temperature for about five minutes, deoxidizer added and the heat cast immediately into a small iron mold. The melting record of heats containing oxide inclusions is shown in Table I.

Table I
Melting Record of Heats Containing Oxide Inclusions

Heat No.	Iron Used	Amount and Type of Deoxidizer Added
1	ingot iron	2.25 per cent ferromanganese
2	electrolytic	3.33 per cent ferromanganese
3	electrolytic	0.16 per cent ferrosilicon (78 per cent)
4	electrolytic	0.27 per cent ferrosilicon
5	ingot iron	0.30 per cent ferrosilicon
6	ingot iron	0.75 per cent ferrosilicon
7	electrolytic	1.00 per cent ferrosilicon
8	electrolytic	1.00 per cent ferrosilicon
9	ingot iron	1.25 per cent silicomanganese
10	electrolytic	1.33 per cent silicomanganese
11	ingot iron	0.30 per cent zirconium-ferrosilicon
12	electrolytic	0.40 per cent zirconium-ferrosilicon
13	ingot iron	0.25 per cent calcium silicide
14	ingot iron	0.25 per cent calcium-silicon-aluminum
15	ingot iron	0.25 per cent aluminum
16	electrolytic	0.33 per cent aluminum
17	electrolytic	0.33 per cent aluminum
18	electrolytic	0.33 per cent aluminum
19	electrolytic	1.66 per cent ferrochromium

The inclusions were studied in the steels in the cast, forged, quenched and annealed conditions. In this study photomicrographs

¹The figures appearing in parentheses refer to the bibliography appended to this paper.

are used to show the various types of inclusions that are formed by different methods of deoxidation. In some cases it is quite impossible to show all that can be seen under the microscope because it is difficult to reproduce color shades. No attempt will be made to describe the few unusual inclusions that were found; the descriptions given apply to the typical inclusions.

SAMPLING AND POLISHING

Transverse sections for metallographic studies were cut from each ingot at a height slightly above the center. A segment was cut from each section and polished for examination. The lower half of each ingot was forged to determine the effect of various inclusions on the forging properties of the iron, as well as to determine the effect of forging on the inclusions.

The method of polishing finally adopted was essentially that described by Urban and Schneidewind (10). The modified procedure is as follows:

1. Grinding on coarse and fine emery wheels.
2. Grinding on French emery paper, starting with a number 1 and finishing with a number 000. Numbers 0 to 000, respectively, were coated with stick graphite, which produced a finer finish.
3. Polishing on a billiard cloth disk, using coarse rouge as an abrasive and water as a lubricant.
4. Polishing on a disk covered with a good grade of moistened billiard cloth, which is charged with a fine grade of jeweler's rouge. Water is dropped on the wheel at a constant speed, the water acting as a lubricant.
5. Washing the specimen in benzene and touching up on the same wheel, using only water.

The relative ease or difficulty with which various specimens polish depends largely upon the types of inclusions present in the iron. With the exception of alumina inclusions, most of the specimens can be polished with little difficulty. Alumina inclusions have a marked tendency to wipe, resulting in the formation of a large number of very fine scratches on the area adjacent to the inclusions.

The various types of inclusions resulting from the reaction of dissolved iron oxide with the added elements in the above heats are described in the following paragraphs.

MANGANESE OXIDE-IRON OXIDE

Heats 1 and 2. Deoxidized with Ferro-Manganese. From the similarity in both chemical and physical properties of FeO and MnO it would be expected that the inclusions formed in Heats 1 and 2 would form a simple solid solution. Herty's experimental work (11) on this system indicates a simple solid solution over the entire range of compositions. Wohrman prepared very pure inclusions which consisted of at least two distinct phases. A number of inclusions were found in Heats 1 and 2 that showed the presence of light and dark gray phases but the majority of the inclusions appeared to consist of only one phase. These inclusions were similar to those shown by Wohrman and no photomicrographs are shown here.

Angular but noncrystalline, dark gray inclusions were also found in limited numbers. These inclusions are usually connected by a series of spherical inclusions of the same color. Körber (12) found that the angular type of inclusion is formed when the MnO/FeO ratio is high, while the round type is formed when the ratio is low. All of the inclusions found in these heats showed a marked tendency to elongate upon forging.

IRON OXIDE-SILICA (FERROUS SILICATES)

Heats 3, 4 and 5. Partly Deoxidized with Silicon. A typical inclusion of Heat 3 is shown in Fig. 1. The most striking characteristic of this inclusion is its larger size. The inclusion shown is very typical in that no other type was found in this heat.

In order to gain some notion as to the composition of the phases of the inclusion shown in Fig. 1, inclusions of this type were removed for petrographic-microscopic studies by a method which will be described in detail in a later section of this paper. The dark irregular areas in this inclusion are glassy in appearance and have an index of refraction that is near that of quenched cristobalite. The semi-glassy ground mass, which is very finely dispersed, is a submicroscopic mixture of silica and fayalite. At first thought it may appear strange that cristobalite would be found in inclusions whose total silica content is low. However, Grieg (13) has shown that nearly pure silica will separate rapidly from a less siliceous mass even when the silica content of the latter is low.

Heating to 1000 degrees Cent. (1830 degrees Fahr.) produced

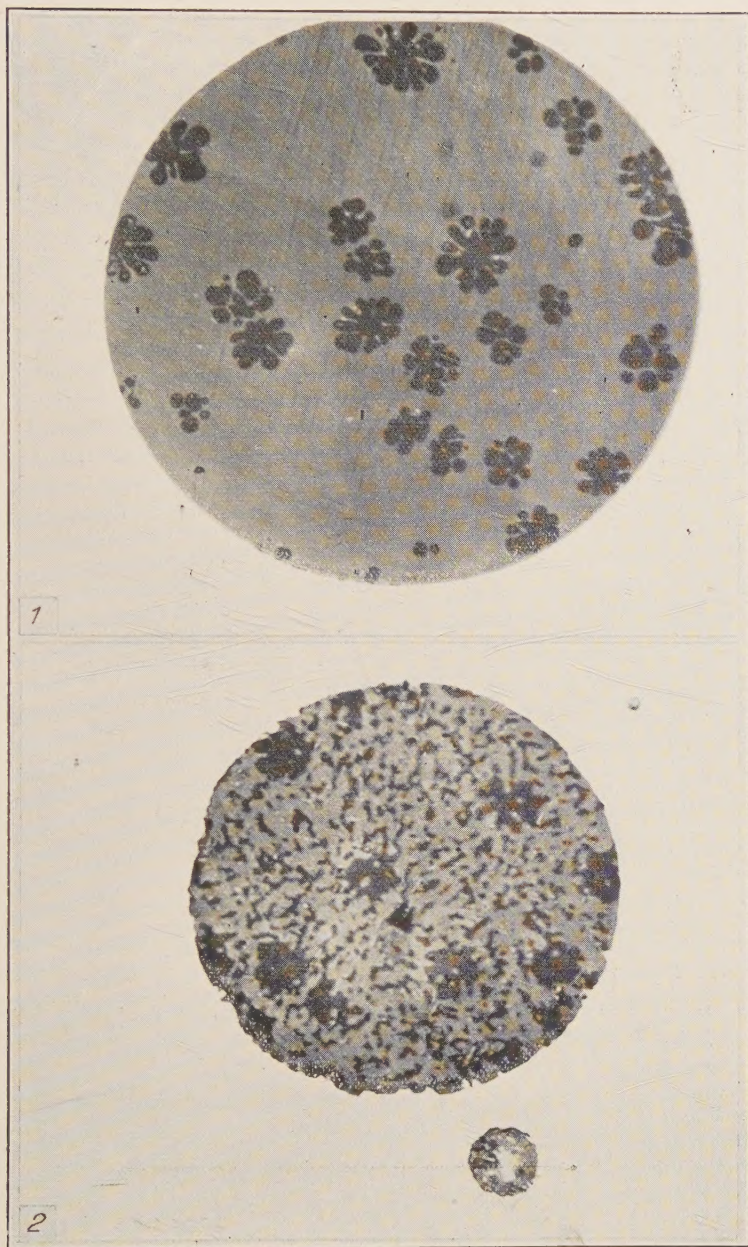


Fig. 1—Ferrous Silicate Inclusion in Heat 3, As-cast; Cristobalite in a Ground-mass of Semi-glassy Silicate. $\times 1000$.

Fig. 2—Ferrous Silicate in Heat 3, Annealed. $\times 1000$.

a distinct structural change in these inclusions. Fig. 2 illustrates the change that has occurred on annealing. The silica particles within the inclusions have lost their original outline; however, they are larger and more numerous, indicating a depletion of the less siliceous ground-mass. That a depletion of the ground-mass has occurred was shown by the fact that it was completely opaque when immersed in the oils used for the refractive index studies.

Since the ground-mass of these inclusions is low in silica it would be expected that their melting point should be comparatively low. This was confirmed by the observation that they readily deform on forging as is shown by Fig. 3.

Typical inclusions of Heat 4 are shown in Fig. 4. This heat had an addition of only 0.27 per cent of ferrosilicon, which is an increase of 0.10 per cent over the addition that was made to Heat 3. This rather small increase in the silicon addition was sufficient to effect a change in size and structure, which is shown by comparing Figs. 1 and 4. The majority of the inclusions found in Heat 4 resemble the gray glassy type shown in the photomicrograph; a few of the inclusions were of the semi-glassy type. The "yeast cell" inclusion suggests coalescence; however, it is equally probable that it may represent an arrested stage of subdivision. These inclusions are high in silica, which is indicated by their low indices of refraction.

It is to be noted that the inclusions shown in Fig. 4 have a characteristic series of concentric rings, which are due to reflections of light from the depths of the transparent spherical inclusion. Wohrman (2) suggested that these reflections originate at the surface of a hole from which the inclusion has been torn out during the polishing operation. Even if, as Wohrman suggests, the cohesion between the inclusion and the metal is very weak, it would still be impossible to dislodge an inclusion that had been cut through a plane above its center. As a final proof that the inclusion is actually present, it may be stated that these inclusions could be gouged out under the microscope.

The inclusions in Heat 5 were quite similar to those found in Heat 4; however, they appeared to be more glassy and were definitely smaller, indicating again that the size of the inclusion is partly governed by the amount of silicon added. The more glassy appearance of the inclusion would suggest that they were higher in silica than the inclusion found in Heat 4. However, the indices of refraction of the inclusions in both of these heats are near that of pure quenched

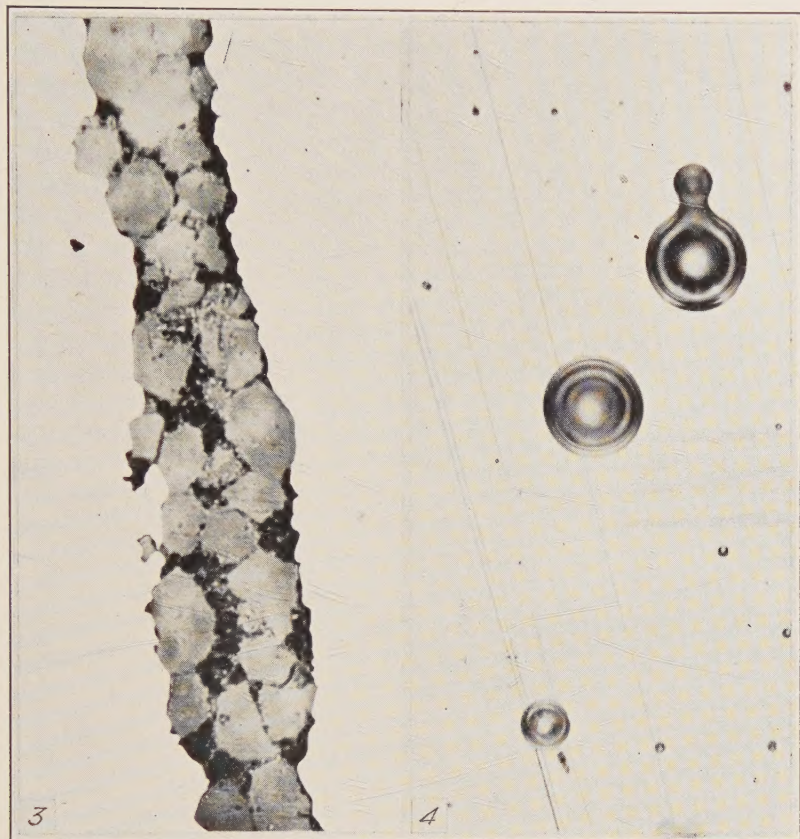


Fig. 3—Ferrous Silicate in Heat 3, Forged. As in the Preceding Photomicrographs the Dark Portions are Cristobalite. $\times 1000$.

Fig. 4—Glassy Silicates in Heat 4, As-cast; Polishing Scratches Indicate Hardness of the High Silica Inclusions. $\times 1000$.

silica. None of the inclusions found in these two heats were affected by either quenching or annealing; however, they were rather easily deformed upon forging.

Heats 6, 7, and 8. Deoxidized with Excess Silicon. Two distinct types of inclusions were found in this series of heats. These are the plain transparent type of inclusions that has been described and the glassy type shown in Fig. 5. The irregular darker areas are similar to the irregular areas shown in the inclusion of Fig. 1, and are pure silica (cristobalite). The ground-mass of these inclusions is nearly pure silica, which was checked by their indices. The trans-

parent, silica-rich ground-mass produces a rather confusing effect on account of the fact that a number of the pure silica particles are located on various planes below the polished section.

A majority of the inclusions in these heats were slightly affected by quenching and annealing, which produced a slight opalescence. This is a rather interesting situation because we have seen that the low silica inclusions of Heat 1 were markedly affected by either quenching or annealing, while inclusions intermediate in silica (Heats 4 and 5) were not affected. The explanation of this behavior is not evident and should be left open for future study.

MANGANESE OXIDE-SILICA. (MANGANOUS SILICATES)

Heats 9 and 10. Deoxidized with Silico-Manganese. The inclusions in these heats could readily be mistaken for those shown in Fig. 5. They contained pure silica embedded in a semi-glassy to glassy ground-mass. The index of refraction of this ground-mass is much higher than that of ferrous silicates rich in silica and is very near that of rhodonite ($\text{MnO} \cdot \text{SiO}_2$). The large size of these inclusions indicates that they would be eliminated from the steel bath with comparative ease. Herty and co-workers (14) have found this to be true of heats deoxidized with silico-manganese alloys.

ZIRCONIUM-FERROUS SILICATE

Heats 11 and 12. Deoxidized with Zirconium Ferrosilicon. All of the inclusions found in these heats were complex in structure; they all showed a green tinge which is characteristic of zirconium silicates. Deoxidation with an alloy containing zirconium would suggest the possibility of forming zirconia (ZrO_2) inclusions, which would be crystalline because the melting point of zirconia is much higher than of iron. These were conspicuous by their absence. Similarly, it may be said that the inclusions are not zircon ($\text{ZrO}_2 \cdot \text{SiO}_2$), nor the eutectic of zircon and zirconia because these also have very high melting points. This leaves only one possibility, namely, that the inclusions formed in these two heats consist of zircon fluxed with either ferrous oxide or ferrous silicates, and would, therefore, be liquid at steel melting temperatures which is indicated by the fact that the inclusions were readily elongated on forging. An example of a large zirconium-ferrous silicate inclusion which has been elon-

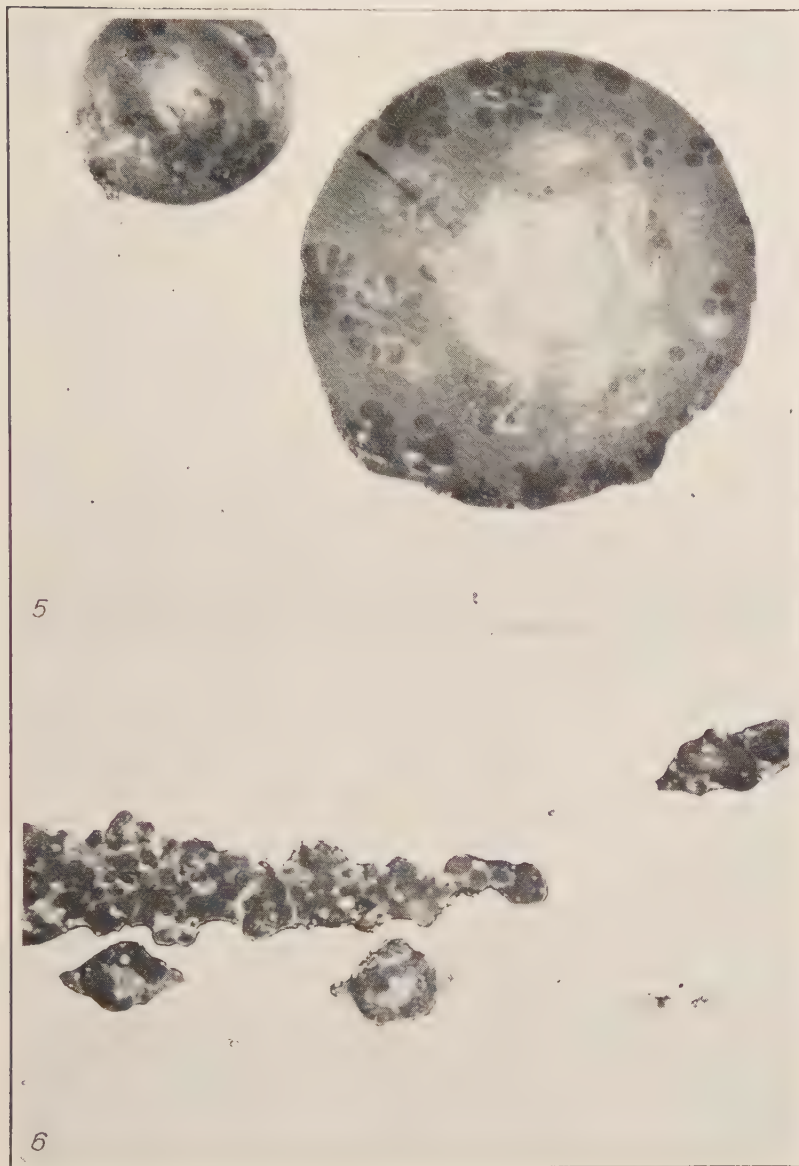


Fig. 5—Glassy Silicates Containing Cristobalite in Heat 8, As-cast. $\times 1000$.

Fig. 6—Zirconium-ferrous Silicates in Heat 12, Forged; Showing the Tip of a Large Elongated Inclusion. $\times 1000$.

gated in forging is shown in Fig. 6. This inclusion was unusually large and only one end of it is shown in the photomicrograph.

COMPLEX SILICATES

Heat 13. Deoxidized with Calcium Silicide. Heat 14. Deoxidized with Calcium-Silicon-Aluminum. The inclusions found in these heats were quite complex, showing as many as three phases, although a number of glassy inclusions were found that were rich in silica and had the same index of refraction as the ferrous silicates high in silica. These glassy inclusions were readily devitrified on annealing. Since the inclusions are so complex in structure it is quite hopeless to try to identify the various phases, however, it may be stated that no free calcium ferrites were found and none of the recognized compounds of the lime-alumina-silica system. The typical inclusions of these heats showed a decided tendency to elongate on forging, indicating that their melting points are below that of liquid steel.

OBSERVATIONS IN POLARIZED LIGHT

When an anisotropic substance is observed in polarized light with the Nicol prisms crossed, the intensity of the reflected or transmitted light depends upon the orientation of the substance relative to the plane of polarization of the light. In the ore microscope used in this study, light passes through the polarizing prism to the specimen from which it is reflected through the analyzing prism to the eyepiece. If the orientation of the specimen is altered by rotating the stage of the microscope, the light intensity varies through successive maxima and minima. When an isotropic crystal is observed with the prisms crossed the intensity of the light is very low and is unaffected by the orientation of the specimen. It is thus possible to distinguish between inclusions which are isotropic and those which are anisotropic without removing them from the steel specimen in which they are found. This method has proven especially useful in the identification of certain sulphides and of the oxides described in the following paragraphs. It is also useful on occasions for distinguishing between sulphides and silicates in rolled or forged specimens. The latter appear brilliantly illuminated against a dark background when examined under crossed Nicols.

ALUMINA AND ALUMINATES

Heats 15, 16, 17, and 18. Deoxidized with Aluminum. The inclusions in these heats are particularly interesting because they have been the subject of numerous discussions. In 1915 Comstock (15) showed by photomicrographs the inclusions that are formed when a bath is deoxidized with aluminum. He described the inclusions as being very small and very difficult to polish, showing a marked tendency to segregate into groups which formed stringers when the steel was forged. Recent studies have not added materially to the description given by Comstock, which may be partly accounted for by the fact that a number of inclusion types are formed under very similar conditions.

In Fig. 7 is shown a typical group of inclusions that were found in Heats 15 and 16. The dark angular inclusion at the top of the photomicrograph was quite rare for these heats, while the irregular groups in the central portion of the photomicrograph are most typical. The irregular groups are very difficult to polish without the formation of "comet tail" scratches, a few of which are still visible even though extreme care had been exercised in preparing the specimen. Numerous investigators have called the irregular dark areas alumina; this is believed to be in error, since alumina should be translucent. The light particle that is embedded in the dark mass may be alumina because under the microscope it is nearly transparent.

Figs. 8 and 9 show the various types of inclusions that were found in ingot 18, which was not dead killed and showed a slight porosity. The gray inclusions shown in Fig. 8 were found near the skin of the ingot. They are ferrous aluminate in various stages of fluxing with the excess ferrous oxide that was present. The inclusions found in the center of the ingot are shown in Fig. 9. Examination in polarized light discloses the fact that the crystals are isotropic and, therefore, not corundum, which is hexagonal. They appear to belong to the cubic system, even in the case of the one crystal whose outline is hexagonal. Now if an iron-alumina spinel were formed, it would be a cubic crystal of the composition $\text{FeO}:\text{Al}_2\text{O}_3$. Such a compound might reasonably be expected, but the work of Herty and his associates (4) on the system ferrous oxide-alumina shows no such compound and indicates that a mixture corresponding to this composition has a melting point considerably below the temperature at which these crystals were formed.



Fig. 7—Typical Inclusions in Heat 15 Deoxidized with Aluminum. $\times 1000$.
Fig. 8—Ferrous Aluminate Inclusion Fluxed with Excess Ferrous Oxide, Heat 18, As-cast. $\times 1000$.

The best we can do at the present time, therefore, is to call these inclusions ferrous aluminate and leave to future research the task of determining their exact nature.

None of the inclusions found in any of the heats deoxidized with aluminum showed any structural change on quenching, annealing, or forging, indicating that all of the inclusions have a rather high softening temperature; for we have seen that oxide inclusions that have a comparatively low melting point are affected by these operations.

CHROMIC OXIDE-IRON OXIDE. (CHROMITE)

Heat 19. Deoxidized with Ferrochromium. Fig. 10 shows the only type of inclusion that was found in this heat. These inclusions are characterized by their purple color and angular form, which is in agreement with the description given by Comstock (15). The crystallinity of these inclusions indicates that their melting point is above that of iron. The same type of inclusions were found in ferrochromium and in chromium steels. As would be expected they are not affected by either quenching or annealing. Since these inclusions have a tendency to segregate into groups like alumina inclusions, they form stringers on forging, although the individual inclusions are not deformed.

Previous investigators have invariably called these inclusions chromic oxide. An examination of Fig. 10 reveals that the inclusions are cubic sections. When examined in polarized light by the method described above they are found to be isotropic. They are, therefore, not chromic oxide, which is hexagonal, but chromite ($\text{FeO} \cdot \text{Cr}_2\text{O}_3$), which is cubic.

PETROGRAPHIC EXAMINATION OF SILICATE INCLUSIONS

The determination of the optical properties of a transparent or translucent substance is not a tedious process once the proper technique is acquired. The methods have been developed by petrographers and are among the most important tools of the mineralogist.

Hartmann (16) studied large inclusions with the aid of polarized light. These inclusions were readily visible to the unaided eye, most of them being stringers which were removed from forged or rolled steels and were about two inches in length and three to five

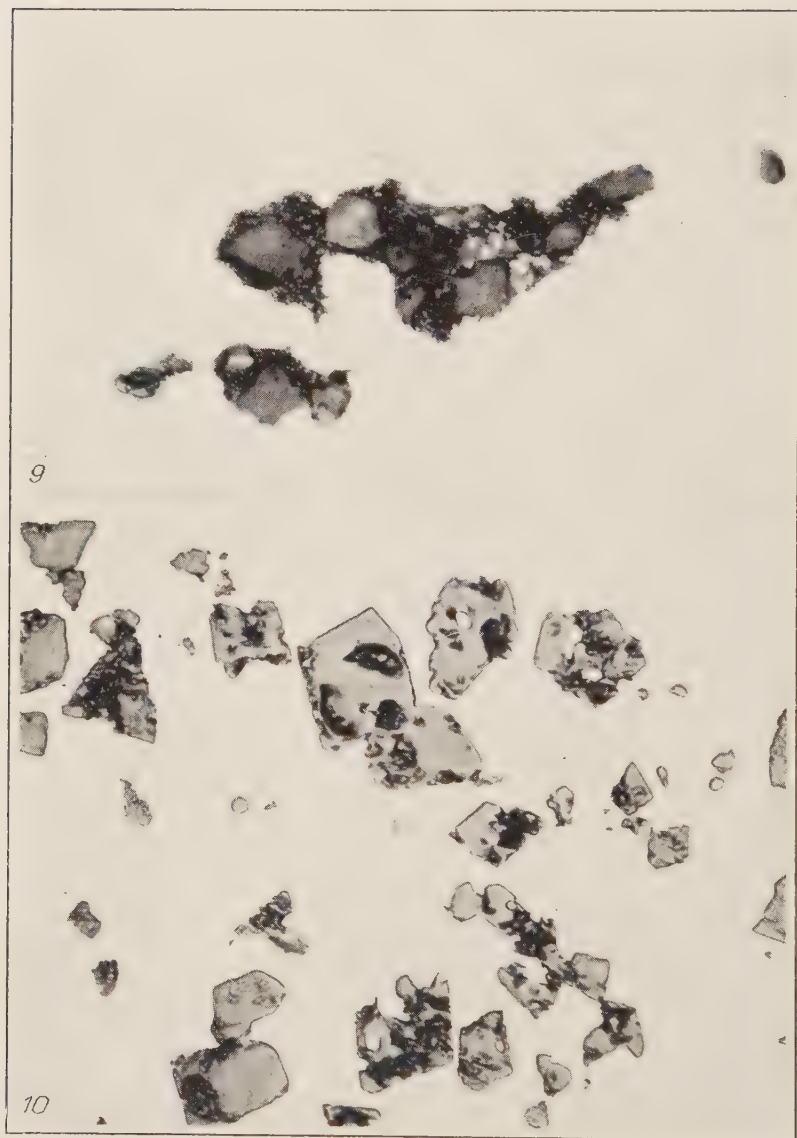


Fig. 9—Ferrous Aluminate Inclusions (Crystals) Held Together by a Material Whose Nature is as Yet Unknown. Heat 18, As-cast, $\times 1000$.

Fig. 10—Chromite Inclusions in Heat 19, As-cast. $\times 1000$.

hundredths of an inch in width. It is evident that inclusions of this size are rare and that Hartmann's technique would not be very useful for the study of ordinary inclusions. From a piece of steel in which an inclusion was embedded Hartmann machined a small plate or rod, containing the inclusion. The separation of the iron was accomplished by the use of chlorine. The steel containing the inclusion was heated at 500-600 degrees Cent. (930-1110 degrees Fahr.) in a chlorine atmosphere, the iron being volatilized as ferric chloride. The residue was then heated in a muffle to remove the carbon. If the inclusion was sufficiently large a thin section was prepared, but if the particles were small or had been obtained by scraping, they were mounted in Canada balsam, sectioned and polished in the usual manner. The identification was made by comparing the slide containing the unknown inclusion with slides of known nonmetallic substances.

The following technique has been developed for the removal of small inclusions and their petrographic examination. In order to be able to remove any particular inclusion that may be of interest for study it is necessary to have a proper tool. This is made by mounting a fine steel needle in the barrel of a watchmaker's screw driver. The needle point appears to be quite large under the microscope; it is, therefore, necessary to grind the needle to an extremely fine point. A specimen is now examined under the microscope and small circles are drawn around a number of the inclusions that appear to be of the same structure. The inclusions are removed by forcing the needle vertically into the steel at a point near the inclusion. By holding the needle point in the hole formed it is a simple matter to dislodge the inclusion by moving the free end of the needle downwards. If the needle is now moistened with oil the inclusion can be picked up and transferred to a slide on which a drop of the oil (of known index) has been placed.

When a substance immersed in oil is observed under the microscope, a band of light appears at the interface between the oil and the substance. As the tube of the microscope is raised this light band (the Becke line) always shifts across the interface in the direction of the substance of higher index. The index of refraction of the extracted inclusion is determined by immersing it in liquids of known indices and noting the movement of the Becke line. The operation is repeated with different liquids until one is found with an index equal to that of the substance at which point the Becke line dis-

appears. This method was found to be especially useful for the silicate inclusions that were not differentiated by the microchemical methods which will be described later.

Ferrous Silicates. The inclusions in Heat 3 are gray in color and of the semi-glassy type. The irregular areas shown in the inclusions (Figs. 1 and 3) have an index of 1.47 which is very close to that of quenched cristobalite. The index of the ground-mass is somewhat greater than 1.74, indicating that it is very low in silica. The quenched and annealed specimens were not suitable for studies because the inclusions were nearly opaque.

The refractive indices of the inclusions in the five heats deoxidized with ferrosilicon are given below. Indices were determined in the cast, quenched annealed states, the two latter being indistinguishable.

Heat No.	Type	Index of Refraction		Remarks
		As Cast	Annealed	
3	Figs. 1 and 3 (ground mass)	1.74	at 1000° Cent. opaque	Low silica
3	Fig. 1 (Precipitated particles)	1.47		Cristobalite
4 and 5	Fig. 4 Glassy	1.47-1.50	1.47-1.50	Silica Glass
6	Glassy	1.53	1.55	Ferrous silicate glass
7	Glassy	1.50	1.50	Ferrous silicate glass
8	Glassy	1.50	1.50-1.52	Ferrous silicate glass

The increase in refractive index in Heats 6 and 8 on heat treatment denotes an increase in the ferrous oxide content of the glass. This was indubitably the result of a precipitation of silica, probably as cristobalite, which was further evidenced by a slight opalescence after annealing. The same devitrification was observed in Heat 3 as shown in Fig. 2. In this case, however, the inclusion after heat treatment was too opaque for a determination of its index.

Manganese Silicates. Most of the inclusions in Heats 9 and 10 contained irregular patches whose index was close to 1.49, indicating that these portions are nearly pure silica. The ground-mass of the inclusions had an index close to 1.72. The indices of refraction of rhodonite ($\text{MnO} \cdot \text{SiO}_2$) and tephroite ($2\text{MnO} \cdot \text{SiO}_2$) are close to 1.73 and 1.78 respectively. Ferrous silicates of an index of refraction of 1.72 would be nearly opaque and, since the inclusions in this heat are glassy, it is apparent that they are manganese silicates, probably containing rhodonite. Annealing

completely changed the structure of the inclusions and the index of refraction was greater than 1.74, which was the limiting value for the oils available. The complex silicate inclusions in Heats 11, 12, 13, and 14 were not suitable for studies because all of them were opaque.

SUMMARY

A series of melts was deoxidized with various elements in order to study the types of inclusions formed. The inclusions studied are nearly pure oxides. All of the inclusions were examined in the steels in the cast, quenched, annealed and forged conditions. A limited number of inclusions were affected by quenching or annealing. These were ferrous silicates low in silica and manganese silicate inclusions. Aluminate and chromite inclusions were not affected by any of the treatments.

Small additions of silicon will produce ferrous silicate inclusions, while larger additions of silicon produce inclusions that are nearly pure silica. Segregated portions of pure silica are found in highly fluxed inclusions whose total silica content is low. It has been further shown that precipitation of silica within these inclusions occurs at 1000 degrees Cent. (1830 degrees Fahr.).

From studies in polarized light it is concluded that the inclusions formed when chromium is added to a steel bath are chromite and not chromic oxide. Similarly it is found that in steels deoxidized with aluminum the inclusions are ferrous aluminates whose exact composition cannot be stated with certainty.

A method for the removal of individual small inclusions has been developed and a petrographic method has been suggested for the classification of silicate inclusions. The method is both rapid and accurate and is not open to the objections that are common to chemical methods.

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